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RUST-INHIBITED NONREACTIVE PERFLUORINATED POLYMER GREASES

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20. ABSTRACT (Cont'd)

lubricating properties comparable to soap-thickened greases with a significant improvement in extreme pressure properties, and are nonreactive at high impact energies in the presence of LOX. The results of this work are applicable to all liquid-fueled rocket engines for missiles and space vehicles.

Rust-Inhibited Nonreactive Perfluorinated Polymer Greases

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Perfluoroalkylpolyether fluids thickened with polytetrafluoroethylene were studied in connection with the development of rust-inhibited chemically inert greases for liquid-fueled rocket engines. It was found that 1.0 to 3.0 wt. percent of a physically and chemically modified organophilic dimethyloctadecylbenzyl ammonium bentonite + sodium nitrite imparts very effective rust-preventive properties to perfluoro polymer grease mixtures. Data are given which show that the rust-inhibited greases are nonreactive on contact with conventional fuels and oxidizers, exhibit lubricating properties comparable to soap-thickened greases with a significant improvement in extreme pressure properties, and are nonreactive at high impact energies in the presence of LOX. The results of this work are applicable to all liquid-fueled rocket engines for missiles and space vehicles.

INTRODUCTION

Perfluorinated alkylpolyether fluids prepared either by photooxidation of perfluoroolefins at −30 C (*1, 2*) or by the anionic polymerization at temperatures below −27.5 C of hexafluoropropylene epoxide in the presence of solvents using cesium fluoride (*3*) have recently been made commercially available. These fluids have considerably lower vapor pressures than the perfluorotrialkylamines which had previously been utilized as components of liquid-fueled rocket engine lubricants (*4, 5*). A recent study of the physical and chemical properties of the alkylpolyether fluids thickened with polytetrafluoroethylene have indicated that the resultant grease mixtures are suitable as inert lubricants for rocket engines powered by liquid propellants such as ethyl alcohol aniline, hydrocarbon fuels (JP-4, JP-5, RP-1), diethylenetriamine (DETA), unsymmetrical dimethylhydrazine (UDMH), hydrazine, hydrogen peroxide, inhibited red fuming nitric acid (IRFNA), nitrogen tetroxide and liquid oxygen (LOX) (*6*). It was found that the polytetrafluoroethylene-perfluoroalkylpolyether grease mixtures exhibited effective lubricating properties, thermal, hydrolytic and oxidative stabilities, wide temperature range, extreme pressure properties, nonreactivity with fuels and oxidizers and shear stability. These lubricants are now extensively used on crew compartment (APOLLO) and launch components (Saturn boosters) of manned and unmanned space vehicles (*7*). While these uses indicate significant progress in the development of chemically inert lubricants for liquid-fueled rocket engines, it has been observed that the polytetrafluoroethylene-perfluoroalkylpolyether grease mixtures do not provide effective protection of ferrous alloys against rust. Using ASTM D1743-64, it was found that lubricated tapered roller bearings, SAE 4620 or SAE 8720 with 1010 mild steel roller retainer, rusted badly after fourteen days exposure at 100 percent relative humidity. A typical illustration is given in Fig. 1.

Further, recent tests (*8*) conducted on a 440C stainless steel R-4 bearing rotating at 3,000 rpm at 5-psi pure oxygen at 70 percent relative humidity lubricated with perfluoroalkylpolyether-polytetrafluoroethylene grease was found to be inoperable after approximately 1,000 hours

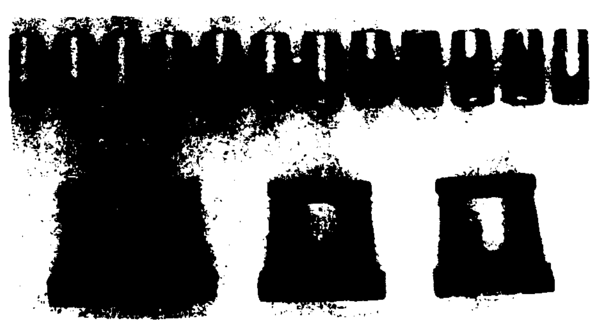

Fig. 1—Uninhibited perfluoroalkylpolyether grease (ASTM D 1743–64)

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due to extreme pitting and rusting. (This bearing is a component of the fan proposed for use for a minimum of 5,000 hours on the astronauts' orbital workshop.)

To overcome this deficiency, work was initiated at the author's laboratory toward the modification of the perfluoro polymer greases with commercially and experimentally available rust inhibitors. Initial exploratory tests (ASTM D 1743-64) indicated that adequate rust protection could not be achieved through the addition of up to 3.0 wt percent of rust inhibitors such as sorbitan monooleate, sorbitan trioleate, barium petroleum sulfonate, fatty amido phosphate and barium dinonylnaphthalene sulfonate. Also, contact compatibility tests with fuels and oxidizers showed evidence of reactivity, and, as a result, such inhibitors could not be considered satisfactory for liquid-fueled systems. In the work described here, a commercially available modified bentonite consisting of dimethyloctadecylbenzyl ammonium bentonite with sodium nitrite (*9*) was found to have rust inhibiting properties with perfluoroalkylpolyether-polytetrafluoroethylene grease mixtures. This paper describes the preparation and properties of a series of rust-inhibited nonreactive perfluoro polymer greases for liquid-fueled rocket motors.

EXPERIMENTAL

Grease Preparation

Materials. The thickener was a low molecular weight tetrafluoroethylene polymer having the following properties: softening point 321.1 C; mol. wt. 20,000–30,000; particle size 100 percent less than 30 microns. The product was supplied as a 7.5 percent suspension in trichlorotrifluoroethane.

The fluids used were fluorinated alkylpolyethers (Table 1). The preparations and properties of these fluids were described previously (*1, 2, 3, 7, 10*).

The additive was a chemically and physically modified organophilic bentonite (dimethyloctadecylbenzyl ammonium bentonite + sodium nitrite) (*9*).

Composition. The compositions of the greases are given in Table 2. All greases were prepared to NLGI Number 2 Grade (265-295 worked penetration).

TABLE 1—PERFLUOROALKYLPOLYETHER FLUIDS

Fluid	Viscosity, cSt. at 37.78 C	Pour Point, C	Density at 23.8 C
PD-1023........	96.3*	−26.1	1.91‡
PD-1024........	153.0*	−29.0	1.91‡
PD-1025........	424.0*	−17.7	1.92
PD-1026........	18.0†	−56.2	1.86
PD-1027........	85.0†	−42.2	1.89
PD-1028........	270.0†	−34.5	1.90
PD-1029........	495.0†	−28.8	1.91

* Ref. (*1, 2, 10*).
† Ref. (*3, 7*); ‡ at 15.5 C.

TABLE 2 GREASE COMPOSITION (WT., PERCENT)

Grease	Perfluoroalkyl-polyether	Thickener‡	Rust Inhibitor§
PD-1030......	85.1(96.3 cSt)*	13.9	1.0
PD-1031......	85.3(153.0 cSt)*	13.7	1.0
PD-1032......	84.7(424.0 cSt)*	14.3	1.0
PD-1033......	85.0(18.0 cSt)†	14.0	1.0
PD-1034......	84.7(85.0 cSt)†	12.3	3.0
PD-1035......	82.3(270.0 cSt)†	15.5	2.2
PD-1036......	87.4(495.0 cSt)†	11.6	1.0

* Ref. (*1, 2, 10*); † Ref. (*3, 7*); ‡ polytetrafluorethylene, mol wt. 20,000–30,000, (*7*); § dimethyloctadecylbenzyl ammonium bentonite + sodium nitrite (*9*).

Dispersion Procedure. Each grease mixture was prepared as follows: The dispersion of PTFE in trichlorotrifluoroethane was heated on a steam bath until ~50 percent of the solvent evaporated. Approximately 75 percent of the required quantity of base oil was then added, the mixture was stirred and heating continued until all of the trichlorotrifluoroethane had evaporated. (The absence of trichlorotrifluoroethane was determined by gas chromatography using the following technique. A sample of the grease mixture was extracted using C.P. benzene. Gas chromatography was used to show (absence of a retention peak after 3.3 minutes) that all the trichlorotrifluoroethane had evaporated (20-ft carbowax 20M column at 50 C, helium flow, 10 ml per minute).

The remainder of the base oil was then added while stirring, and stirring continued until a homogenous grease-like product was obtained. The mixture was cooled to room temperature. The rust inhibitor was then added, thoroughly mixed into the grease, the mixing being completed by passing the grease twice through a colloid mill with a stator-to-rotor clearance set at 0.001 inch. The homogenized mixture was permitted to remain at room temperature for 24 hours prior to use. Greases were prepared in 200-g batches (Table 2).

Rust Preparation

Initial studies were directed toward establishing the minimum quantity of rust inhibitor required for each grease to pass the rust test using the tapered roller bearing described in ASTM D1743-64. (This test was used since it is currently specified in numerous specifications, e.g., MIL-G-23827, MIL-G-25013, MIL-G-21164 and MIL-G-81322). The minimum quantity of rust inhibitor required for rust protection is given in Table 2. The same test was run on grease samples prepared without the inhibitor and also on uninhibited commercial greases (*7, 10*). Figures 1 and 2 are typical of the test results; all bearings lubricated with greases containing the rust inhibitor showed no corrosion; all samples without the inhibitor rusted badly. This was found to be so whether the viscosity of the fluid used to prepare the test grease was as low as 18.0 cSt or as high as 495.0 cSt at 37.78 C (Table 2).

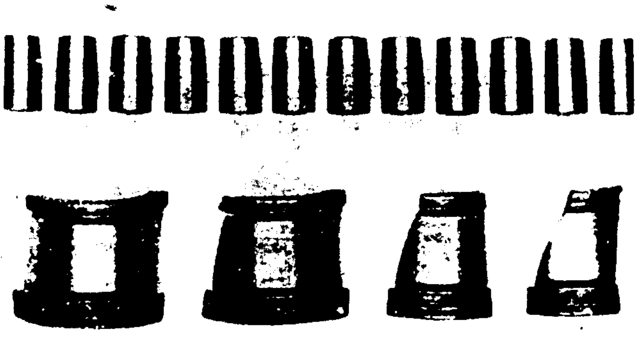

Fig. 2—Inhibited perfluoroalkylpolyether grease (ASTM D 1743-64)

Reactivity with Fuel and Oxidizers and Metals at High Shear

Contact Tests. Contact compatibility tests were run at 25 ± 1.0 C. One gram each of the material being tested (or 1 ml if fluid) was placed in a 5 ml graduated glass-stoppered cylinder and 1 ml of fuel or oxidizer added. Visual observations preceded by shaking were made after five minutes and 1, 24, 48 and 72 hours. The fuels and oxidizers used were EtOH, JP-4, UDMH, DETA, $C_6H_5NH_2$, N_2H_4, H_2O_2, IRFNA and N_2O_4. The tests using N_2O_4 were run in closed pressure glass jars 1 x 5 inches. The results (Tables 3, 4) show that the low molecular weight polytetrafluoroethylene thickener was nonreactive and insoluble with all of the test fuels and oxidizers. The dimethyloctadecylbenzyl ammonium bentonite + sodium nitrite rust inhibitor swelled but showed no reactivity. The data in Tables 3 and 4 indicate no reactivity of the test materials with the fuels and oxidizers. Although some of the fuels and oxidizers were somewhat soluble in the perfluoroalkylpolyether fluids, the degree of solubility was less on the greases made up with the same base oils. Only a slight solubility (< 5 percent by volume) of the grease was noted. Evidently the structure of the grease dispersion is such that the fluid is not readily accessible for solution.

Impact Tests. Liquid oxygen impact compatibility tests were run at an impact level of 72.3 foot-pounds (*11*). The thickener, rust inhibitor, fluids and greases were considered nonreactive with LOX if they withstood 20 separate impact trials without reaction (flashes, explosions or other indications of sensitivity). Since none of the materials tested were found to be reactive with LOX, within the limits mentioned in Table 5, it appears that the thickener, the rust inhibitor, the fluorinated polymer fluids and greases made from these products may be useful for rocket motor systems which use LOX as the oxidizer. It is significant to note that the modified organophilic bentonite was not sensitive when tested alone (PD-1016) or as part of the grease mixtures (PD-1031, PD-1034 and PD-1035) when subjected to high-impact energy levels. PD numbers in text and tables are in-house code designations for experimental materials used by the author's laboratories.

Metals at High Shear. It has been reported that explosive reactivity occurs when aluminum surfaces are coated with polytetrafluoroethylene (*12*) or polymers of chlorotrifluoroethylene (*13-16*) when subjected to mutual shear at high loads. To determine whether similar reactivity is associated with the products described here, tests were run on the fluids and greases using proposed ASTM D-2 Method (*17*). Seven tenths ± 0.1 of a ml of materials was placed in a cylindrical hole (½-in dia x ½ inch deep) in a block of 2024-T4 aluminum. A dowel of 2024-T4 aluminum (¼-in diameter, rounded end (0.250 ± 0.001-inch radius tip) × 3.0 inches long) was rotated into the block at 1,760 rev per min under a load of 1,000 psi for one minute. The dowel mating surface in the speci-

TABLE 3—CONTACT COMPATIBILITY OF THICKENER, RUST INHIBITOR AND BASE FLUIDS

MATERIAL	EtOH	JP-4	$C_6H_5NH_2$	UDMH	DETA	N_2H_4	H_2O_2	IRFNA	N_2O_4
PD-821*	I	I	I	I	I	I	I	I	I
PD-1016†	I	I	I	I	I	I	I	I	I
PD-1024‡	I	I	I	~10	~10	W	I	~10	~20‖
PD-1027§	I	I	I	~ 5	I	W	I	~ 5	~15‖
PD-1028§	I	I	I	I	I	W	I	~ 5	~10‖

* Thickener; † rust inhibitor; ‡ Ref. (*1, 2, 10*); § Ref. (*3, 7*); ‖ fuel or oxidizer soluble in base oil, vol, %; W, white emulsion; I, no apparent change.

TABLE 4—CONTACT COMPATIBILITY OF GREASES

GREASE*	EtOH	JP-4	$C_6H_5NH_2$	UDMH	DETA	N_2H_4	H_2O_2	IRFNA	N_2O_4
PD-1030	I	I	I	SS	SS	SS	SS	SS	SS
PD-1031	I	I	I	I	I	I	I	SS	SS
PD-1032	I	I	I	I	I	I	I	I	SS
PD-1033	I	I	I	I	SS	SS	I	SS	SS
PD-1034	I	I	I	SS	I	I	I	SS	SS
PD-1035	I	I	I	I	I	I	I	SS	SS
PD-1036	I	I	I	I	I	I	SS	SS	SS

* Composition given in Table 2; I, no apparent change, SS, slightly soluble (<% 5.0 percent by volume).

Table 5—IMPACT COMPATIBILITY

Material	LOX#
PD 821*	Not reactive
PD 1016†	Not reactive
PD 1024‡	Not reactive
PD 1027‖	Not reactive
PD-1028§	Not reactive
PD-1031	Not reactive
PD-1034	Not reactive
PD-1035	Not reactive

* Thickener; † rust inhibitor, ‡ base oil Ref. (*1, 2, 10*); § base oil Ref (*3, 7*); ‖ grease; # no reaction in 20 trials Ref (*11*).

men block was made using a fluted carbide-tipped ball-end end mill 0.500 ± 0.001-in in diameter with 0.250 ± 0.001 in radius tip finished to 8 to 16 microinch rms. The load is weighted to provide a 1,000 psi at the dowel and specimen block mating surfaces. The data is given in Table 6. All of the lubricants tested were reactive to some degree: none appeared to be as sensitive as the polychlorotrifluoroethylene fluid (*13–16*). The addition of the rust inhibitor did not increase the reactivity of the lubricant with aluminum at the 1,000 psi load. However, since the data is important in the potential use of the lubricants for metallic connectors and thread fasteners in rocket motor systems, it would appear that as a precautionary measure this apparent deficiency of the lubricants should be checked on any batch contemplated for use.

Table 6—REACTIVITY OF LUBRICANTS WITH ALUMINUM AT HIGH SHEAR*

Lubricant	Explosive Reactions No of Trials
Perfluoroalkylpolyether, (96.3 cSt)†	4/6
Perfluoroalkylpolyether, (96.3 cSt) + thickener	1/6
Perfluoroalkylpolyether, (96.3 cSt) + thickener + rust inhibitor	1/6
Perfluoroalkylpolyether, (85.0 cSt)‡	2/6
Perfluoroalkylpolyether, (85.0 cSt) + thickener	1/6
Perfluoroalkylpolyether, (85.0 cSt) + thickener + rust inhibitor	1/6
Perfluoroalkylpolyether, (270.0 cSt)‡	2/6
Perfluoroalkylpolyether, (270.0 cSt) + thickener	1/6
Perfluoroalkylpolyether, (270.0 cSt) + thickener + rust inhibitor	1/6
Polychlorotrifluoroethylene oil, 6.5 cSt at 37.78 C	6/6

* Load, 1000 psi at 1760 rpm, dowel and block 2424-T4 Al, Ref. (*17*); † Ref. (*1, 2, 10*); ‡ Ref. (*3, 7*).

Lubricant Properties

Antiwear characteristics were determined on the four-ball wear tester, using ASTM D 2266-64T. Wear scar diameters were measured on the three stationary balls after one hour at 1200 rpm at 75 C with 10-kg and 40-kg loads using a travelling microscope at 40 X. The values are the average of the readings taken parallel and normal to the scuff marks. The data (Table 7) shows that the addition of the rust inhibitor to each thickened oil lowers the scar diameter to slight degree.

Extreme Pressure (EP) properties of the test greases were determined using the four-ball method described in ASTM D2596-69. It was found that the rust-inhibited perfluoro polymer greases did not weld under an applied load of 800 kg.

Comparisons were made with similar greases containing no rust inhibitor. The data (Table 7) show a marked improvement in EP properties due to the addition of the rust inhibitor. Further, the EP characteristics of the inhibited or uninhibited polytetrafluoroethylene-perfluoroalkylpolyether greases are far superior to the currently used EP MIL SPEC greases, e.g., MIL-G-23827, MIL-G-21164, or MIL-G-81322.

It is also of interest to note that the scar diameters on the balls in the EP tests using ASTM D 2596-69 which did not weld at 800-kg loads were all below 4.0 mm. These data (no weld at 800-kg load or scar diameters below 4.0 mm) clearly suggest that the modified organophilic bentonite plays a major role in enhancing the EP properties of the test greases.

Other Lubricant Properties. The data (Table 8) show that the inhibited perfluoro polymer greases exhibit high dropping points, low water washout characteristics, low

Table 7—ANTIWEAR AND EXTREME PRESSURE PROPERTIES

	Antiwear*		Extreme Pressure†	
	Wear Scar Diam, mm			
	10-Kg Load	40-Kg Load	Weld, Kg	Scar Diam, mm
Base Oil, 96.3 cSt‡ + thickener	0.418	1.235	400	...
Base Oil, 96.3 cSt + thickener + rust inhibitor	0.275	1.095	>800	2.383
Base Oil, 85 cSt§ + thickener	0.462	0.796	800	3.533
Base Oil, 85 cSt + thickener + rust inhibitor	0.297	0.603	>800	2.218
Base Oil, 270.0 cSt§ + thickener	0.502	0.943	600	...
Base Oil, 270.0 cSt + thickener + rust inhibitor	0.301	0.632	>800	2.761
MIL-G-23827	...	...	<300	...
MIL-G-21164	...	...	<400	...
MIL-G-81322	...	...	<300	...

* ASTM D 2266-64T; † ASTM D 2596-69; ‡ Ref. (*1, 2, 10*); § Ref. (*3, 7*).

TABLE 8—LUBRICANT PROPERTIES

GREASE	DROPPING POINT, C*	BLEEDING WT, %†	WATER WASHOUT WT, %‡	EVAPORATION WT, %§	SHEAR STABILITY UNWORKED, NO STROKE	10,000 STROKES	100,000 STROKES
PD-1031	318(281)‡	4.5(4.8)	1.5(1.3)	0.21(0.36)	269	324	312
PD-1034	280(281)	3.1(2.4)	2.2(3.7)	0.93(0.03)	271	365	305
PD-1035	272(275)	2.4(2.6)	2.3(3.9)	0.07(0.06)	296	317	324

* ASTM D 2265-67; † Fed. Std. Test Method 321.2 (100 C for 30 hr), ‡ ASTM D 1264-63 at test temperature of 38.0 ± 3.0 C; § ASTM D972-56; ‖ ASTM D 1403-62; ¤ Data in parentheses are values on greases without the rust inhibitor

TABLE 9—TORQUES (G-CM) ON PERFLUORO GREASES*

GREASE	INHIBITED STARTING	INHIBITED RUNNING	UNINHIBITED STARTING	UNINHIBITED RUNNING
PD-1031 (1.0)†	5074‡	1770‡	3540‡	1925‡
PD-1034 (3.0)†	1307§	1032§	4071§	2478§
PD-1035 (2.2)†	8053‡	2212‡	5768‡	1667‡

* ASTM D 1478-63; † rust inhibitor, wt, %; ‡ test temperature, -22 C, § test temperature, -40 C.

vapor pressures, and good mechanical stabilities. It is of interest to note that no adverse changes took place in these properties due to the addition of the rust inhibitor.

Limited tests were also conducted to determine the effect of the rust inhibitor on low temperature torque. It was found (Table 9) that the starting torques of the inhibited greases were higher than the uninhibited greases. It should be noted, however, that the torque values of the inhibited perfluoro polymer greases compared favorably with the products meeting MIL-G-27617 "Grease, Aircraft, Fuel and Oil Resistant." The latter specifies a 7,000 g-cm starting torque at —22.0 C.

SUMMARY OF RESULTS

The primary purpose of the work was to develop rust-inhibited nonreactive perfluoro polymer greases for liquid-fueled rocket motors. Based on the test data given, it must be concluded that the modified ammonium bentonite imparts effective rust-prevention properties to perfluoroalkylpolyethers fluids thickened with polytetrafluoroethylene. It is further concluded that the rust inhibitor is nonreactive (alone or in grease mixtures) on contact with the fuels and oxidizers (Tables 3, 4) and under impact with LOX at high energy levels (Table 5). The lubricating properties of the inhibited polytetrafluoroethylene-perfluoroalkylpolyether greases are comparable to soap-thickened greases with a marked improvement in extreme pressure properties (Tables 7–9). The lubricants reported here are applicable to all liquid-fueled rocket engines for missiles and space vehicles.

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